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ABSTRACT

This teacher's guide on nuclear science is divided into two parts. The first part is a discussion of some of the concepts in nuclear chemistry including radioactivity, types of disintegration, radioactive decay and growth, and tracer techniques. The relevant experiments involving the use of radioisotopes are presented in the second part. The experiments include the use of Geiger counters, investigations of the properties of radiations, and separation techniques such as paper chromatography, ion exchange, solvent extraction and precipitation. A simple circuit diagram of a Geiger counter is included. This handbook is intended primarily for teachers of secondary school science in Asia. (LC)

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CHEMISTRY TEACHING PROJECT

IN ASIA

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EXPERIMENTS ON NUCLEAR SCIENCE

SE 008 164

1969

BANGKOK, THAILAND

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PREFACE

This handbook is intended as a teacher's guide on nuclear science. Since this topic is not quite as generally known as other topics of chemistry, the handbook is therefore divided into two parts. The introduction to nuclear science covers the normal requirements for those who have basic scientific knowledge, but wish to employ radioisotope techniques in their work. The practical approach to the use of radioisotopes has been made through various topics such as radioactive growth and decay, methods of detection of radiations, tracer techniques. The relevant experiments are presented in the second part. Since this handbook is primarily prepared for the benefit of the science education at the secondary school level where apparatus, equipments, chemicals and isotopes are difficult to obtain, the experiments have therefore been designed for simplicity but are adequate in a way of illustration of the basic properties of radiations and the applications of radioisotopes. Teachers are encouraged to use natural occurring isotopes such as uranium and potassium and also to purchase or even make a simple kind of counter. For the latter purpose, a simple circuit diagram of a GM-counter is included in the second part of the book. The author acknowledges the Unesco Chemistry Teaching Project's staff and the Thai Atomic Energy Commission's staff for their technical assistance. The Electronic Section of the Thai Atomic Energy Commission has succeeded in producing the inexpensive counters. Most of the experiments appearing in this handbook have been tested using one of these counters.

The contents in this handbook do not claim to be original or complete in any sections; additional reading is advisable for those who want to be specialised in the field of nuclear chemistry. Constructive criticisms on this work are sincerely welcome.

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PART I

INTRODUCTION TO NUCLEAR CHEMISTRY

INTRODUCTION TO NUCLEAR CHEMISTRY

The main difference between chemical reactions and nuclear reactions is that chemical reactions involve the rearrangement of electrons in the orbit outside the atomic nucleus whereas nuclear reactions involve the changes within the nucleus. It is therefore essential for the study of nuclear science to start from the study of the nucleus.

1.1 The Nucleus and Radioactivity

An atomic nucleus is the central core of an atom and is composed of 2 types of particles, protons and neutrons. These particles possess approximately the same mass (mass of proton = 1.007280 a.m.u., mass of neutron = 1.008665 a.m.u.) but the proton has a positive charge whereas the neutron is neutral. It is obviously seen that an atomic nucleus carries a positive charge. In the nucleus, particles are bound together by a nuclear force. Each species of nucleus contains a definite number of protons and neutrons. The nuclear charge is balanced by extra nuclear electrons. These three are the fundamental particles which are constitution of atoms. There are other sub-atomic particles such as mesons, neutrinos, anti protons etc, but they need not concern us here. We shall use the symbols Z, N and A for an atomic number (the number of protons in a nucleus), a neutron number (the number of neutrons in a nucleus) and a mass number (the sum of Z and N) respectively. In this article the convention ${}_{Z}(\text{chemical symbol})^{A}$ e.g. ${}_{15}P^{32}$ will be used to indicate the characteristics of nuclei. Three types of nuclides are classified using these 3 numbers as criteria.

Table I

	Z	A	N	Examples
Isotope	same	different	different	${}_{15}P^{31}$ and ${}_{15}P^{32}$
Isobar	different	same	different	${}_{15}P^{32}$ and ${}_{16}S^{32}$
Isotone	different	different	same	${}_{1}H^{2}$ and ${}_{2}He^{3}$

The chemical properties of an atom are due almost entirely to the electrons rather than to the nucleus. Hence from Table I the isotopes of a given element have the same nuclear charge and the same number of extra nuclear electrons. Therefore, they will have virtually identical chemical properties but slightly different physical properties such as mass, density, freezing point. If it were not for some tiny differences due to the different masses of their nuclei, it would be impossible to separate isotopes from each other by chemical means.

The sum of the mass of component particles in the nucleus is nearly equal to the actual mass of the nucleus in atomic mass units; the difference is small and it is due to the binding energy for the nucleons in the nucleus. The binding energy is the energy which would be required to split the nucleus into its component

nucleons and is usually expressed as binding energy per nucleon.

Not all combinations or ratios of protons to neutrons are stable, if there is an excess or deficiency of neutrons, the nucleus will spontaneously disintegrate in order to reach a more stable arrangement, in other words the atom will be radioactive. The radioactive atom is unstable, its nucleus undergoes disintegration all the time with characteristic half-life (see later). It is also characterised by the type and energy of the emitted radiation. The process of disintegration is affected by the temperature, pressure or concentration of the reacting substances. There are 3 kinds of radiations emitted from the unstable nuclei, alpha(α), beta (β) and gamma (γ) radiations. These radiations can affect photographic plate so that they are darkened on development, they can ionise a gas by ejecting an electron from an atom of gas and produce scintillation or small flashes of light in certain substances such as zinc sulphide. The magnetic field can deflect alpha and beta particles but not gamma rays. Alpha particles are the nuclei of helium atoms travelling at high speed and have a low penetrating power (a thin sheet of paper or a few centimetres of air can stop alpha particles completely). They produce intense ionisation in the few centimetres of air. Beta particles are fast-moving electrons and can penetrate a few millimetres of aluminium or about 700 centimetres in air. Gamma rays are electromagnetic radiation like x-ray but have shorter wavelengths or higher frequencies. They can penetrate long distances through air or several centimetres through lead.

1.2 Types of Disintegration

a. Alpha Disintegration. Usually alpha particles are emitted by heavy elements e.g. radium emits alpha particles and decays to radon. Most isotopes emit particles of more than one energy. Magnetic analysis of alpha particles gives a spectrum consisting of one or more sharp lines, each line corresponding to a particular kinetic energy. Suppose two nuclei of U^{238} decay by emitting alpha particles of different energy to form 2 nuclei of Th^{234}. According to the first law of thermodynamics, the Th^{234} produced by the emission of the lower energy α-particles from U^{238} has more energy than that obtained from the higher energy particle emission.

The more energetic nucleus is called an excited stete of Th^{234}. In order to come to a lower state of energy (more stable), Th^{234} must emit the extra energy in the form of an electromagnetic radiation, a gamma ray.

b. Beta Disintegration. Ordinary beta decay, as in the natural radioactive series, is commonly found throughout the range of the periodic table. This phenomenon occurs when there is an excess of neutrons in a nucleus. In order to achieve a stable state, one neutron splits into a proton and an electron. Only the negative electron (negatron) with high speed is emitted. This particle is termed β^-. The emission of beta particles differs from that of alpha particles in respect to the spectrum of the energies of the emitted particles. A characteristic feature of the

spontaneous beta disintegration of a nucleus is the continuous distribution in energy of the emitted electrons from zero up to a definite maximum energy. Thus, it is reasonable to adopt Pauli's suggestion that another particle with zero mass and zero charge, must escape simultaneously with the beta particle, carrying off a variable fraction of the available energy. This particle is termed neutrino and it was proved by experiments that it really exists. Some beta decays may first produce an excited state of product nucleus followed by the emission of a gamma ray in order to get to a ground state of energy.

Fermi suggested that both e^- and e^+ (see below) and neutrino are actually created at the moment of decay.

c. Gamma Emission. The gamma rays produced from a radioactive product give a line spectrum corresponding to a number of discrete frequencies in the emitted radiation. Gamma radiation originates in the atomic nucleus, while x-radiation originates from the orbital electrons. It should be noted that gamma rays are emitted by the daughter nucleus and not the parent nucleus. Usually they are emitted immediately after the alpha and beta particles. However, when there is a delayed gamma ray transition from an excited state to a lower energy state, it is called an isomeric transition and is regarded as a pure gamma emission, e.g. $Br^{80m} \xrightarrow{\gamma} Br^{80}$.

d. Positron Emission. The positron is the positive electron (e^+) whose rest mass is the same as that of the electron and whose charge has the same magnitude as that of the electron but the opposite sign. This type of disintegration occurs where there is a deficiency of neutrons. Positron emission results in a decrease by one unit in atomic number.

$$_7N^{13} \longrightarrow {}_6C^{13} + e^+$$

As an alternative to emitting a positron, some nuclei capture a negative electron from one of the atomic electron orbits nearest to the nucleus. The electrons which are most likely to do this are those nearest to the nucleus i.e., K shell; less often L-electrons are captured e.g.

$$Cu^{64} + e^- \longrightarrow Ni^{64} + \nu(\text{neutrino})$$

There is no nuclear radiation except for the unobservable neutrino from a pure K or L capture process. The secondary process is that an electron from an outer shell jumps to fill the vacancy in K or L shell immediately with the emission of a K or L x-ray characteristic of the daughter nucleus. This process is called Electron Capture and occurs when the amount of energy available in a nucleus is less than 1.02 MeV.

e. Nuclear Fission. A heavy nucleus is broken down into two medium heavy fragments with about the same mass. The fission of a nucleus is accompanied by a release of energy, since the binding energies of the product nuclei are greater. The most important difference between fission decay and the other types of

decay is that the masses and energies of the products of fission are all statistically distributed.

Nucleons are bound together in a nucleus by nuclear force and this force tends to keep the nucleus in spherical shape (the most stable configuration for a heavy nucleus). At the same time the Coulomb force between protons tends to make the nucleus unstable and this force is sufficient, in the heaviest nuclei, to upset the stability of the nucleus. It was found that this process happens to the nuclei whose atomic numbers are near or over 100. So it can be seen that it is possible for spontaneous fission to occur, and it does indeed take place in a number of the heaviest nuclides.

Usually fission is induced by bombardment of nuclei with bombarding particles, preferably neutrons. For example

$$_{92}U^{235} + {}_{0}n^{1} \longrightarrow {}_{92}U^{236} \longrightarrow {}_{Z}X^{Ax} + {}_{Z}Y^{Ay} + 2.5\ {}_{0}n^{1} + \text{energy}$$

$_{Z}X^{Ax}$ and $_{Z}Y^{Ay}$ are fission products. The fission products were found to have mass numbers from 70 to 160.

The important nuclides strontium-90 and cesium-137 are found during the process of nuclear fission. The energy liberated in this process is just equivalent to the loss of mass. One unit of mass on the C^{12} atomic weight scale is equal to 1.66032×10^{-24} gm, which, from Einstein's equation, corresponds to 931.437 MeV.

The main modes of disintegration are summarised in Table II.

Table II

	charge	mass	Resultant nucleus	Example
Alpha particle (α), nucleus of He (range in air = 0.5 cm for 1 MeV particle)	+2	4	Z decreases by 2, A decreases by 4	$_{92}U^{238} \xrightarrow{\alpha} {}_{90}Th^{234}$
Beta particle (β), electron originated from nucleus (range in air can be as much as several metres)	-1	0	Z increases by 1, A remains constant	$_{90}Th^{234} \xrightarrow{\beta} {}_{91}Pa^{234}$
Gamma (γ), electromagnetic wave (very penetrating)	0	0	change in energy level only	$_{35}Br^{80m} \xrightarrow{\gamma} {}_{35}Br^{80}$

1.3 Radioactive Decay. The disintegration of radioactive nuclei is a purely random process. The decay of radioactive isotope is a first order reaction, i.e. the decrease per unit time in the number of atoms of a radioactive element, due to disintegration, is proportional to the number of atoms which have not yet disintegrated:

Number of decaying per second = a proportionality constant characteristic of the isotope times the number of nuclei remaining

In the notation of the calculus, this is

$$-dN/dt = \lambda N \qquad \text{----------(1)}$$

Where N is the number of nuclei remaining, and λ is the proportionality constant for the isotope. It is called the decay constant. If at some particular time there were N_0 nuclei in the sample, then we can find an expression for N, the number remaining at any later time t, by integration of equation (1). The result can be expressed in various equivalent mathematical forms:

$$N = N_0 e^{-\lambda t} \qquad \text{----------(2)}$$

$$\ln(N/N_0) = -\lambda t \qquad \text{----------(3)}$$

$$\log_{10} N = \log_{10} N_0 - 0.4346\lambda t \qquad \text{---------(4)}$$

At any instant of its life, the nucleus has a definite probability, unaffected by its age, of disintegrating during the next second. The higher this probability, the more rapidly the nuclide may be expected to disintegrate. If there is a 1% probability of disintegration in the next second, it is not possible to state which particular nuclei will undergo this process, but one can only say that 1% out of a very large number of such nuclei will decay during this time interval.

It is not possible to speak of the total life of a number of atoms. A useful constant is the "half life", the time needed for half of the radioactive atoms to disintegrate. After one half life (i.e. after $t=t_{\frac{1}{2}}$ and $N/N_0 = \frac{1}{2}$) equation (3) becomes

$$\ln \tfrac{1}{2} = -\lambda t_{\frac{1}{2}}$$

$$\text{or } 2.303 \log \tfrac{1}{2} = -\lambda t_{\frac{1}{2}}$$

$$t_{\frac{1}{2}} = \frac{0.693}{\lambda}$$

It can be seen that equation (4) is the equation of a straight line. The half-life can be found from the plot of log N against t. If the plot is not linear, there is more than one radioactive nuclide present. The half-life for any particular nuclide is a characteristic constant. The determination of half-life is often

used for characterising a particular nuclide. The values of half-life vary, from one nuclide to another, from a small fraction of time to many millions of years.

1.4 Activity

An activity of 1 curie simply means that 3.7×10^{10} disintegrations occur each second, irrespective of the radioactive nuclide concerned. More convenient units are the millicurie (10^{-3} curies) and the microcurie (10^{-6} curies). It is important to realise that the activity of a sample refers to the number of disintegrations per second, and not to the count rate recorded with a particular piece of equipment. Count rate depends on the overall efficiency of the counting system and the number of radiations emitted per disintegration (e.g. sometimes two or more gamma ray photons are emitted in each disintegration).

1.5 Radioactive Growth

If radioactive nuclide "A" decays into a nuclide "B" which is also radioactive and this is in turn disintegrate to "C" which is stable we have,

$$A^* \xrightarrow{\lambda_A} B^* \xrightarrow{\lambda_B} C$$

When the rate of formation of B^* is equal to the rate of disintegration of B^* to C (or the rate of formation of "C"), the state is called equilibrium.

There are three special cases which are worthy of interest.

a. Secular Equilibrium. Here the parent A has a much longer half life than B. It can be shown that the activity of B becomes approximately constant after a time equal to 5 half-lives of B and the total activity of the preparation of A and B any time is the sum of the initial activity of daughter at that time. For example in the series

$$_{92}U^{238} \xrightarrow[t_{1/2}\ 4.5\times10^9\text{yrs}]{\alpha} {}_{90}Th^{234} \xrightarrow[t_{1/2}\ 24.5\text{ days}]{\beta} {}_{91}Pa^{234}$$

$$\beta \downarrow \ t_{1/2}\ 1.14\text{ min.}$$

$$_{92}U^{234} \xrightarrow[t_{1/2}\ 2\times10^6\ \text{yrs}]{\alpha} {}_{90}Th^{230} \longrightarrow$$

All the members of this series have shorter half life than that of U^{238}, therefore in the uranium compound or uranium ore left unprocessed for a long time so that equilibrium has had time to be established, all members of the series are in radioactive equilibrium with the U^{238} and with each other. This radioactive equilibrium presents a considerable practical application for example as a source of relatively short lived radioactive tracers (like Pa^{234}) for chemical, biochemical and physical application by process of "milking" them as required.

b. Transient Equilibrium. The parent is longer lived than the daughter but sufficiently small for decay to be noticeable within the time of observation. In this case the activity of B* decreases with the characteristic half life of A*. For example

$$Ba^{140} \xrightarrow[t_{1/2}\ 12.8d]{\beta,\ \gamma} La^{140} \xrightarrow[t_{1/2}\ 40\ hrs.]{\beta,\ \gamma} Ce^{140} \quad \text{(stable)}$$

c. There is no equilibrium when the parent has a shorter half life than the daughter ($\lambda_A > \lambda_B$). If the parent is separated from its daughter in the beginning, as the parent decays the number of daughter atoms will rise, pass through a maximum value and after the parent has decayed, the daughter is no longer formed, and will decay with its own half-life. For example

$$Ce^{143} \xrightarrow[t_{1/2}\ 33\ hrs.]{\beta} Pr^{143} \xrightarrow[t_{1/2}\ 13.7d.]{}$$

1.6 Tracer Techniques

Radioactive isotopes and their stable ones are essentially chemically identical. In addition, the chemical reaction of the former can be traced by means of their characteristic half-life or their disintegration product or the properties of the radiation they emitted etc. When the radioactive materials are used instead of their stable isotopes in any conventional chemical techniques, they are called "tracer techniques". Since the weight of radioisotope necessary to give a measurable activity is often less than 10^{-15} grammes, i.e. unweighable amounts, it is therefore possible by using these tracer techniques, to study the behaviour of material in micro, trace, or even ultramicro quantity either qualitatively or quantitatively.

a. Tracers or radioactive indicators. These are radioactive nuclides used to follow the movement of the atoms of certain elements or compounds throughout any mechanical, chemical or biological systems. They can be divided into two types.

(i) Isotopic tracers. Radioactive tracers and their stable isotopes are chemically identical. The tracer and the stable isotope must have the same chemical form so that they can act in the same manner throughout the process. In the system involved with elements of several valency states, it is advisable to make sure that they are in the same form by repeating oxidation-reduction cycles several times. Isotopic tracers are used to trace elements or compounds in question from the beginning to the end of the process, such as in studies of metabolism in the body, diffusion, rate of reaction, analysis etc.

(ii) Non-isotopic tracers or physical labels. These are used when the experiment is not concerned with any particular element or atom. The tracers need not be identical with the stable element. For example, radon gas has been mixed with air to study the flow patterns of air jetted into a furnace to improve combustion. Non-isotopic tracers may be used for locating a solid or

liquid object. They have been used to discover leaks in complicated systems of pipes and tubes.

b. Carriers. These are the stable materials added to the radioactive samples and they act as carriers for the active materials in all subsequent reactions.

From the decay equation, $-dN/dt = \lambda N$, the weight per curie of nuclides of different half-lives may be calculated. In many cases only 10^{-3} microcuries can be detected and measured. Hence, in radiochemical work, the masses involved are very small (of the order 10^{-16} to 10^{-19} grammes) so small so that solubility products cannot normally be exceeded and precipitation is, therefore, impossible. In order to carry out precipitation and reduce effects of adsorption it is necessary to use "carriers". A sample containing no carrier is called "carrier free". There are three types of carriers.

(i) Isotopic carriers. Carriers and radioactive nuclides have the same chemical form and state. This type of carrier is used when high specific activities (disintegrations per unit time per gramme) are not necessary.

(ii) Non-isotopic carriers. A chemically different element is used to carry the radioactive tracer. This type of carrier is used when no isotopic carrier is available or when the sample of high specific activity is needed because this carrier may be separated from an active material at the end of the experiment and pure active material in the carrier free form is obtained.

(iii) Scavenger type. Where no other suitable carrier may exist, adsorption on the surface of a precipitate such as ferric hydroxide, or almost any gelatinous or finely divided precipitate, can be used to carry out precipitation of certain radionuclides.

Most radioactive isotopes for use in medical, agricultural and industrial research are made in the nuclear reactor. The reactor can be regarded as the machine that produces neutrons and an enormous amount of energy as a consequence of the controlled fission process (see fission on page 4). The reactor furnishes a bombardment of neutrons that attach themselves to the nuclei or centres of the atoms of an ordinary element that is called the "target material." The typical examples of the production of radioisotopes by the neutron bombardments of the target materials in the nuclear reactor are given below.

$$_{16}S^{32} + {}_{0}n^{1} \longrightarrow {}_{15}P^{32} + {}_{1}p^{1} \quad \text{or} \quad {}_{16}S^{32}(n,p)\,{}_{15}P^{32}$$

$$_{11}Na^{23} + {}_{0}n^{1} \longrightarrow {}_{11}Na^{24} + \gamma \quad \text{or} \quad {}_{11}Na^{23}(n,\gamma)\,{}_{11}Na^{24}$$

$$_{3}Li^{6} + {}_{0}n^{1} \longrightarrow {}_{1}H^{3} + {}_{2}He^{4} \quad \text{or} \quad {}_{3}Li^{6}(n,\alpha)\,{}_{1}H^{3}$$

1.7 Methods of Detections of Radiations

A very important effect of emission of radiations is to produce ionisation in air and other materials and this ionising effect is the basis of the detection systems. Beta particles and alpha particles produce ionisation directly; gamma rays by the charged secondaries which they release and which are directly measurable. A gold leaf electroscope can be used as a simple detector for the presence of ionising radiations (see later).

Beta particles, alpha particles and gamma rays may be detected by the use of some nuclear detectors which make use of the ability of radiations to ionise air or other gases such as an ionisation chamber, a proportional counter (suitable for the counting of an alpha or a beta particles with low energy), a Geiger Muller counter (suitable for the detection of strong beta emitters), a scintillation counter (suitable for the detection of gamma rays or weak beta emitters, the design of this counter based on the ability of electrons to cause fluorescence in certain substances) in conjunction with an electroscope, a scaling unit or a rate meter.

The ability of the radiations to darken a photographic plate or a film is found useful in the technique called "autoradiography" (see later).

1.8 Operational Problems in Tracer Experiments

According to the report of I.A.E.A./UNESCO Panel of Experts on Nuclear Science Teaching, Bangkok, July 1968, three levels of radiation hazard may be distinguished corresponding to the different educational levels.

Less than 16 years. This may be regarded as the level at which radiation doses are trivial and hardly discernable from daily background. Most schools with science laboratories will be in this class by virtue of chemicals in store or television sets used in teaching. The radiation levels for students experiments should not exceed those recommended in 5.1, 1.3 (ii), (iii), (iv), (v) of the I.A.E.A. Safety series No 9, 1967.

16-18 years. Student experiments with unsealed sources in this age group (and teacher demonstrations to the under 16 group), should be such that activities do not exceed those listed in column 7 of Table II A and B of the I.A.E.A. booklet Safety Series No.9, 1967. Experiments with sealed sources may use higher levels of activities, for example those specified in the booklet issued in the United Kingdom by the Department of Education and Science (document AM 1/65).

It may be desirable to place an upper limit on the total amount of radioactive sources kept in store in any laboratory. Waste disposal is not a problem at these levels of activity and unsealed sources may be disposed of as for ordinary chemical waste.

Above 18 years. In this group, universities and colleges will be

covered either by the levels referred to above, or by licences for higher activity levels issued by national authorities.

Radioactive sources of the type and activities used in this handbook require only normal, good chemical practice for their safe handling. Uranium, thorium and potassium salts are very popular sources of radiations since they are quite safe to work with (normally used as a chemical agent) presenting no radiation hazards. Besides, uranium and thorium salts can be used as a "cow" so that many isotopes can be milked off. Uranium oxide is used regularly as a reference counting source due to the long half life of uranium-238. Monazite sand can also be used as a counting source. The general guide lines for physical considerations are given below.

The experimenter has to consider:

a. Health hazards and contamination control. This needs practice and discipline. Radioisotopes are useful because of the radiations they emit, but at the same time they present a potential hazard to the isotope workers and, in certain circumstances, to the general public. All of us have learned to live with fire and electricity which are dangerous as well as useful, we can therefore learn to live with radiation too. Danger from radiation depends on the degree of exposure. The exposure to radiation can be limited in three ways:

(i) Distance. In general, the effect of radiation falls off with distance; the inverse square law can be applied.

(ii) Shielding.

(iii)Time. Stay in the vicinity as short a time as possible.

b. Nature and energy of the emitted radiation. This is important in relation to the method of detection of the radiation.

c. Half-life. A suitable half-life nuclide is to be chosen for a certian experiment, that is a nuclide of sufficiently long half-life to maintain its activity up to the end of observations, but not so long that disposal problems arise.

d. Chemical procedure. This has to be carefully chosen to suit the properties mentioned in b. and c. of this section.

e. Isotope effect. The chemical identity between isotopes of the same element is not complete among the light element, e.g. H^1 and H^3; the mass differences between these isotopes are great enough to cause different chemical properties. The effect, owing to the large difference in mass, is pronounced in any reaction where mass is involved. Thus, there are changes in rate of reaction, equilibrium constant or bond strength etc. For all elements with atomic mass above carbon this isotope effect is small and can usually be ignored.

f. Dummy run with non-active isotope is always essential.

Most, if not all, of the difficulties concerning manipulations will be solved as known beforehand, hence contaminations and health hazard are minimised.

Radioactive materials should only be used when there is no other equally suitable method available. There are numerous uses of radioisotopes, where the hazard from emitted radiations is great, but can be controlled. Precautions, <u>through understanding</u> of the subject and training will minimize this hazard considerably since to know is to understand and to understand is to act with wisdom.

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PART II

EXPERIMENTAL SECTION

A. COUNTERS, STATISTICS OF COUNTING AND SOURCE PREPARATION

When a radiochemical work is carried cut, it is almost always that the experimenter has to deal with counting by at least one form of counters. Thus it is essential to know about the performance of the counter including counting and the preparation of a counting source.

A.1 Counters

Only a simple Geiger counter with an end window Gieger tube will be discussed here. A circuit diagram of the type used by the author is shown. The G.M. counter is operated at a potential determined by its characteristic curve. The curve is obtained by plotting the count rate due to a fixed and constant radicactive source as a function of voltage across the counter. At a low voltage (before A) the pulses are too small to operate the counting

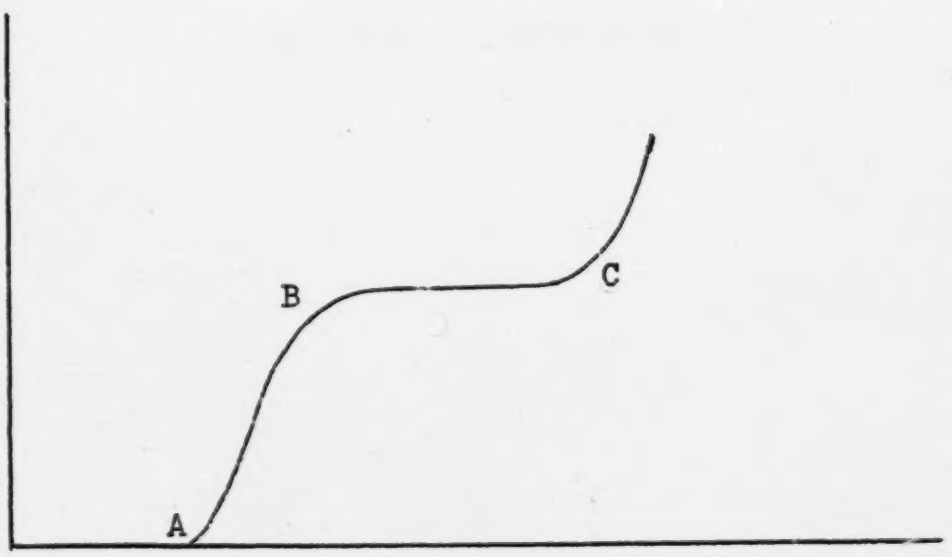

Fig. 1 Characteristic curve of a counter.

equipment The "starting voltage" A is reached where the pulses become large enough to trigger the equipment; and there follows a "plateau" where there is little change in the count rate as the voltage increases over the range say 100-250 volts. Beyond this the count rate shows a very steep rise and the tube may go into continuous discharge. In order to ensure stable operation the counter is worked with an applied voltage roughly corresponding to the centre of the plateau. The first task of the user of any type of counters is to determine its characteristic curve by taking readings from a radioactive source at small voltage intervals until point C in the graph above is reached, results are plotted and the operating voltage is obtained. This should be carried out regularly. However, most of the school type counters are produced in such a way that the users will encounter with a minimum complication

such as the G.M. tube used with such a counter will receive a fixed voltage supply correspond to the operating voltage of that tube, hence there is no need to determine an operating voltage.

A.2 Statistics of Counting

Radioactive atoms decay statistically, that is the time at which one atom will decay is independent of the decay of all other atoms and cannot be predicted. This randomness of the decay of radioactive atoms can and should be illustrated in the class because its consequence concerning errors in counting of activity is very important in radiochemical work. Two suggestions for the illustrations of this property are given below.

1. It can be shown by an analogue experiment such as the famous one; throwing coins. Student throws 100 coins or more collects and counts only those turn tails up, discards those with the opposite faces up or vice versa. Toss the remainder, collect and count those with tails up again and discard the rest. This process should continue as many times as possible. It can be shown from the results that the most probable event is that half of the total coins will turn heads up and the other half turn tails up and the least probable one is that every coin turns only head or tail up. This is applied with a large number of coins and a large number of events. The plot of the logarithm of number of coins at the end of each event against the appropriate eventi.e., first event, second event and so on should give a straight line with a negative slope and this experiment can be used to explain the meaning of exponential decay and half-life.

2. Fix source at a position under a G.M. tube and repeat the counting 50 times or more. Record each count in counts per unit time. Results show that the coincidence of the values of the count rate is really a very rare event. In fact what normally happens is that the number of counts "N" obtained changed statistically about the mean value "$\bar{N}$". Therefore students should be warned that they should not expect the count rate will be exactly the same when they repeat the measurements even under the identical condition and that the randomness of disintegration is responsible for this.

If count rates obtained in the above paragraph are arranged into group with a small intervals of count rate within each group such as 25 or 50 and are plotted on the abscissa and the numbers of count rate that fall into each group are on the ordinate. Under the perfect condition a bell shape curve should be obtained from which a mean count, standard deviations may be read off as illustrated in the graph below.

From statistical consideration, the error of a set of measurements is approximately equal to the square root of the mean count (or of the count rate itself in the case of a single measurement). It immediately follows that

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Total counts	Standard deviation(σ)	Limits of counts
100	$\sqrt{100}$	100±10 (10% error)
1,000	$\sqrt{1,000}$	1,000±31.8 (3.2% error)
10,000	$\sqrt{10,000}$	10,000±100 (1% error)

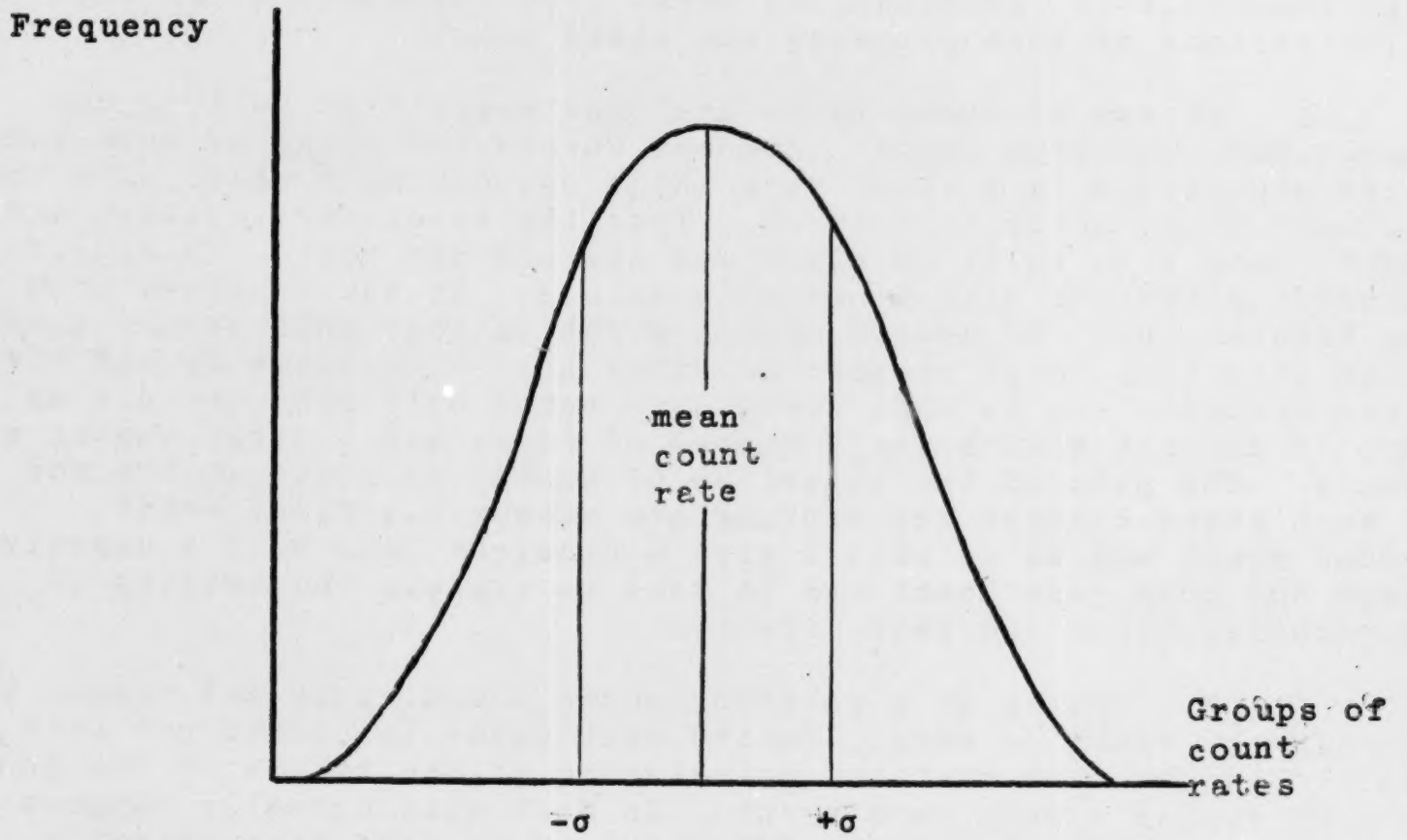

Fig. 2 Statistical distribution of the count rate

It is advisable therefore to collect as many counts as possible in order to reduce the statistical error to a minimum. It is a common practice that the counting time should be long enough to obtain about 10,000 counts. However, it may not be practical in the counting of a very low activity source since one has to wait too long in order to obtain a total count of that order. In such a case it is left to the experimenter to decide about the length of time that can be spent in the measurement of the activity, bearing in mind that the lower the total count the higher is the error of the counting.

A.3 Source Preparation

A reference source, that is a source with a constant activity over many many years due to its long half life, is usually made from uranium compounds. For example, uranium oxide is made a slurry with a little acetone and "Durofix" or "Duco Cement" or the likes and is spread as a thin film over the bottom of a small aluminium or stainless steel source tray about 1 inch in diameter and is dried under an I.R. lamp so that the boiling of the sample is avoided. A thin foil of aluminium is placed on top of the tray and the source is counted at 2 cm below the tube window, about 10,000 - 15,000 counts per minute (c.p.m.) are aimed at. If the first layer does not yield high enough count rate, following layer should be applied successively until the desired count rate is reached. A little of "Durofix" or "Duco Cement" is then smeared around the rim of the tray and the aluminium foil is stuck onto it.

If monozite sand is used as a source of radiations, the above procedure may not be suitable since the activity from the sand is usually not high enough. Instead, a small amount of the sand is put on a small watch glass and the glass is placed immediately under the tube and is counted.

A point source can be made from a solution of the radioisotope concerned. A hypodermic syringe is used to transfer the tracer solution from its container, usually a multidose vial and a small drop is placed at the centre of an aluminium or a stainless steel source tray or a planchette as it is normally called. The source is dried under an I.R. lamp and is checked for its count rate. More drops can be added if a higher count rate is required. Finally the source is sealed with a piece of cellotape.

However, if one wants to spread the radioactive solution evenly over a larger area one can cover the bottom of the tray with a piece of a filter paper circularly cut to fit exactly that part of the tray, and a few drops of a radioactive solution is applied which will spread evenly over that piece of paper and the trayed is covered in the usual manner.

It should be stressed that when making up a counting source from a radioactive substances, surgical gloves should be worn and precautions should be taken as to avoid contamination of the experimenter and the working area. The dispensing of radioisotopes should be carried out on a small tray which is covered with a piece of non-absorbent paper.

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B. PROPERTIES OF RADIATIONS

Radioactive elements undergo spontaneous transformation from one chemical atom into another; this is accompanied by emission of radiations whose basic properties are mentioned on page 3-5 of Part I. The following experiments illustrate various properties of radiations both with and without the aid of a nuclear detector.

Experiment B.1 Deflection of Radiations by Magnetic Field

Alpha, beta and gamma rays are characterised by their behaviours in the electric and magnetic fields. The rays bending towards the left in the diagram carry positive charges and are called alpha (α) rays. Those deflected to the right carry negative charges are called beta (β) rays. The third type gamma (γ) rays go unaffected as they carry no charge at all. The deflection of these rays by the magnet can be shown quite conveniently in the class.

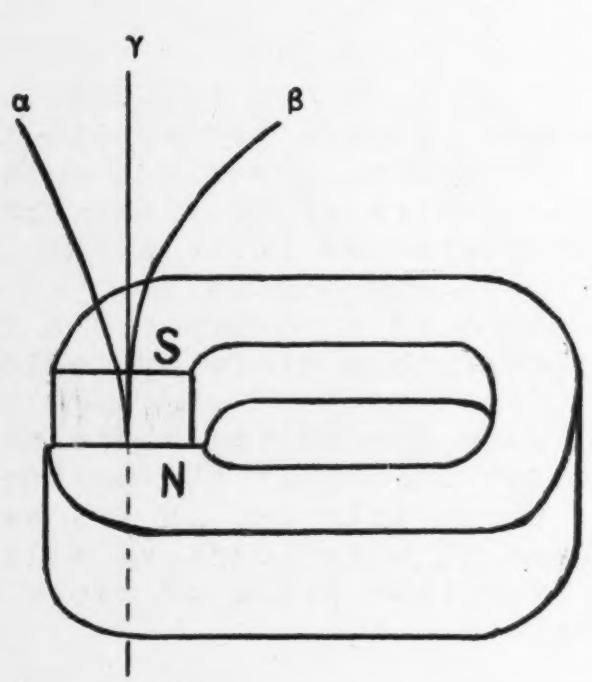


Fig. 3 Deflection of radiations by magnetic field

Materials Required

Alpha source
Beta source } point source should be used.
Gamma source
Horseshoe magnet
Collimator
Detector

Procedure

A beam of radiation to be tested is collimated through a small hole so that the scattering of the beam is minimised and the majority of the radiation emitted from the source will be efficiently detected. The count rate is recorded after which the magnet is placed on top of the collimator (This can be made of any light material in the form of a block with a small hole, of the same size as the source, in the middle through which radiation pases). If the source is a gamma one there will be no change in the count rate but if it is an alpha or beta source, the count rate will be reduced considerably, the remaining count rate is due to background radiation and to the gamma component which always contaminates alpha and beta sources to some extent. Scanning of the G.M.-tube in searching for the deflected radiations reveals that they are deflected as indicated in Fig. 3, and that beta particles are deflected to higher degree than alpha particles owing to its low mass.

Experiment B.2 Ionising Property of Radiation

When a charged particle, passes through matter, it will either excite or ionise the atom; an orbital electron is raised to a state of higher energy within the atom in the former case and ejected in the latter. To demonstrate the effects caused by air ionising materials e.g. flames, glowing wood splinters, hot wire, α-,β-rays, a "home made" electroscope can be used. It is easy to construct by students in the class.

Materials Required

A flask or wide neck bottle with fitted rubber stopper
Gold foil
A thick copper wire or a big screw nail
A copper plate (e.g. door knob)
Plastic comb, ruler or fountain pen
An alpha or beta source.

Procedure

The electroscope consists of a conical flask, into which is fitted a rubber stopper. The stopper carries a thick copper wire or a long screw nail. A big piece of a copper plate such as a door knob is soldered to one end of this wire. The other end of the copper wire (inside the flask) is flattened, and two strips of a gold foil about 1/4 inch wide and 1/2 inch long are stuck on to it. The electroscope is charged with a plastic comb, a ruler or a fountain pen which has been rubbed with a piece of wool or satin. The α-particle source is brought near but not touching. The leaves should slowly come towards each other, showing the α-particles from the source are ionising the air and thus making it a conductor. This can be compared roughly with the effect of heat such as from a lighted match or sunlight.

Ionisation can also render the grain of a photographic emulsion developable. When a photographic film is exposed to ionising radiations, it is blackened to an extent depending on the amount of energy absorbed. The method of detection of radiations makes use of this effect such as in the device called "film badge" which is used in the measuring of the integrated dose that was received during a previous period. Use is also made of this effect of radiation in the technique called "autoradiograph". An example of the application of this technique is given in experiment C.1.2.

Experiment B.3 Absorption

When charged particles (i.e. α-, β-particles and secondary electrons resulting from interaction of γ-rays with matter) pass through matter, they will lose energy through the ionisation they produce. The more they produce ion-pairs the less energy left and thus the less far they can travel. The distance travelled by a charged particle in matter is called "range". In a particular material the energy loss is proportional to the mass per unit area of the absorber e.g. mg/cm^2 (range in aluminium) or g/cm^2 (range in lead). Among the three, α-particles have the lowest penetrating power whereas β-particles have a medium one and γ-rays are the most penetrating of all.

Equal thickness of a given material absorbs the same fraction of the incident γ-radiation i.e., the absorption is exponential and a plot of the logarithm of counts per minute against thickness of the absorber is linear. The validity of the inverse square law ($I \propto 1/d^2$, where I is the intensity of light or radiations and d is the distance) exists in the absorption of γ-rays, but this law does not hold in the β-radiation absorption. The attenuation of β-radiation depends on the density of the material and in most cases is independent of atomic weight. The natural β-spectrum has a definite maximum energy (characteristic of each radionuclide) and, corresponding to this, a maximum range. If a series of an absorbing material are placed between a β-ray source and a detector, it is found that activity of the source detected decreases as the thickness of the absorber is increased up to a limit where no significant activity is recorded at all. While the absorption of β- and γ-rays is quite simple to observe using an ordinary G.M. counter and a set of different thicknesses of the absorber (usually aluminium or lead absorber is employed), the absorption of α-radiation is not at all easy to study with ordinary counter owing to the very short range in matter of α-particles, about half of the alpha energy is used in penetrating the Geiger tube window.

Materials Required

G.M. counter
Alpha source
Beta source (such as P^{32})
Gamma source (such as Cs^{137})
Set of aluminium absorbers with different thicknesses
Set of lead absorbers with different thicknesses

Procedure

A beta source is placed under the G.M. tube and the count rate is recorded. A series of an aluminium absorber with different thicknesses is placed between the detector and the source successively and the count rate is recorded for each one. The count rate should decrease little with a thin foil of aluminium but more when the thickness is increased until there is no appreciable change in count rate with increasing of the absorbers thickness, indicating the β-particles of all energies up to the maximum emitted from the source are absorbed. The minimum absorber thickness required to stop all particles from entering the G.M. tube or any nuclear detector is termed "maximum range" and can be obtained accurately from the graph of log. c.p.m. against thickness of absorber (Fig.4)

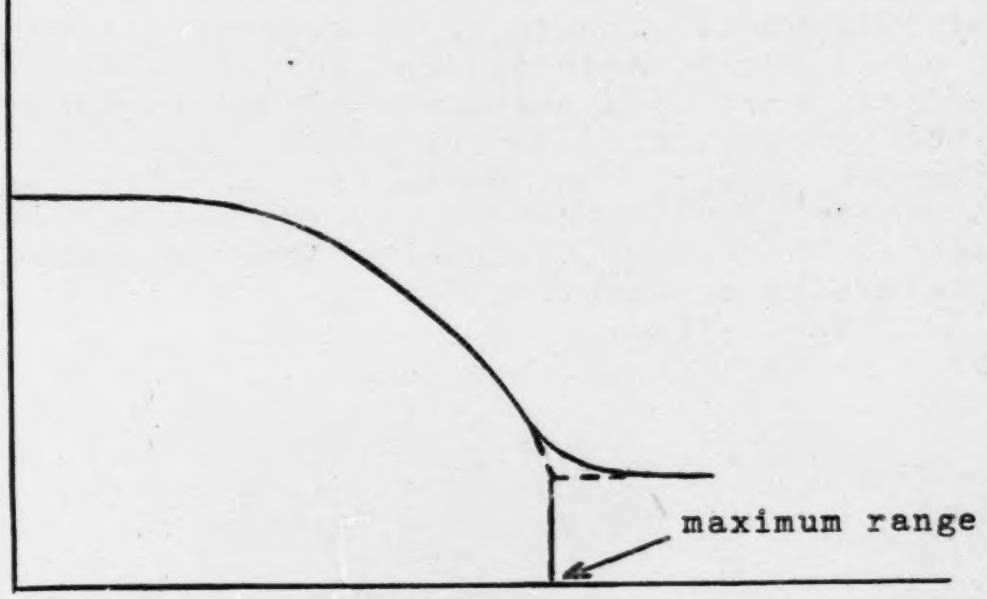


Fig.4 Absorption curve for β-particles

The maximum energy is calculated from the relationship.

E_{max} = 0.00185 R + 0.245 (R is the range in mg/cm^2 and is greater than 300, E_{max} is the maximum energy in Mev). Alternatively, the maximum β-energy may be found from the standard curve of maximum β-energy against maximum range in aluminium.

When the same procedure is employed for the determination of the γ-ray absorption using lead absorbers, the plot of log c.p.m. vs absorber thickness is linear. Hence there is no maximum range, instead, a half thickness is used and it is a characteristic of a particular nuclide. The half thickness is the thickness of lead

absorber needed to reduce the count rate to one half. The corresponding energy of a γ-ray can be read off from the standard curve of half thicknesses against energy.

However, for the pre-university level, it is quite sufficient to carry out experiments as far as to show the existence of the maximum range for a β-emitter and a half-thickness for a γ-emitter and that those values are different for different isotopes.

It is also thought worthwhile to compare the penetrating power of β-and γ-radiations. If the position of the GM- tube is fixed and the position of the source of radiation is varied with respect to that of the tube (1-2 mm at a time). It is found that the order of the distance travelled, in air is $\alpha<\beta<\gamma$.

The inverse square law can be studied for β- and γ-radiations. This time, the positions of the source with respect to that of the tube are known for each count rate recorded. For γ-emitter, a plot of count rate against $1/(\text{distance})^2$; or even better a plot of square root of reciprocal of count rate against distance yields a straight line hence the inverse square law is valid. This is not true for a β-source. It is appropriate therefore to point out to the students that particles of different energies are absorbed by different distances in air. The deviation from the inverse square law for β-radiations is attributed to the fact that this extra effect is added on, otherwise the simple inverse square law of intensity of radiation would be valid.

C. SEPARATION TECHNIQUES

It is quite important for radioisotope users to be familiar with the normal methods employed in the separation of radioisotopes. The recommended isotopes for use in schools are either short lived nuclides or nuclides belonging to the decay chain of uranium and thorium. If the latter type of nuclides are used, one needs to separate the desired nuclide from other members of the chain. The radiochemical purity thus can be checked by the type and energy of the emitted radiations and/or by means of its characteristic half-life. Methods which have been used for clean and efficient separation of radionuclides are as follows: volatilisation, distillation, electrodeposition, precipitation, solvent extraction, chromatography, and ion exchange. The experiments based on some of the above techniques are described below:

C.1 Paper Chromatography

Paper chromatography is also employed in the separation of ions in radiochemical technique as well as in the ordinary analysis. The principle is that when a suitable solvent flows by capillary action along a strip of filter paper, any spots of material which may be on the paper, and which are soluble in the solvent used will travel along the paper in the direction of the solvent flow. The distance travelled is dependent on the partition coefficient (the various components of the original mixture move with the solvent but at different rates). It is customary to calculate the R_f-factor for each separated nuclide. This is defined as

$$R_f = \frac{\text{distance travelled by the spot front}}{\text{distance travelled by solvent front}}$$

R_f-factor of an ion has a characteristic value under the given conditions.

As far as the activity is concerned the microcurie quantity is usually sufficient to be detected radiochemically. The actual number of radioactive atoms present however is so vanishingly small that it will be adsorbed on the paper support and on the walls of the apparatus. To eliminate this, small quantities of the isotopic carrier are usually added.

Experiment C.1.1 Radiochemical Purity of $^{32}PO_4^{3-}$ by Ascending Paper Chromatography.

Material Required

Gas jar + lid + paper holder
Strip of chromatography paper (preferably Whatman No.1)
Strong carrier free $^{32}PO_4^{3-}$ solution
A 4% vol/vol nitric acid in methanol
Carrier solution (10% vol/vol ortrophosphoric acid solution)
Spray solution (5 g ammonium molybdate dissolved in 100 ml water poured into 24 ml nitric acid and 24 ml water with stirring).

Procedure

1. A strip of paper 30 cm long x 3 cm wide is marked out in pencil as follows. A line is drawn across the paper 2 cm from one end and this is called a base line. The paper is now marked out in cm from this base line and numbered.

2. A 4% vol/vol nitric acid in methanol is poured into a gas jar to a depth of 2 cm. The length of the paper strip is then adjusted (outside the jar) so that the base line will be about 1 cm above the liquid level when the apparatus is assembled. The paper should not be wetted at this stage.

3. A drop of carrier free $^{32}PO_4^{3-}$ solution is now applied in the middle of the base line (the minimum of about 5000 counts per minute under a thick window G.M.-tube should be aimed at) and dried under an I.R.-lamp, after which a spot of a carrier solution is added in the same positfon and again is dried.

4. The apparatus is now assembled using a jar lid and paper clips or a piece of "cellotape" to fix one end of the paper on to the lid (Fig. 5). The paper must not touch either sides of the gas jar and is allowed to develop for 2 hours. The position of the alcohol front is marked on the chromatogram.

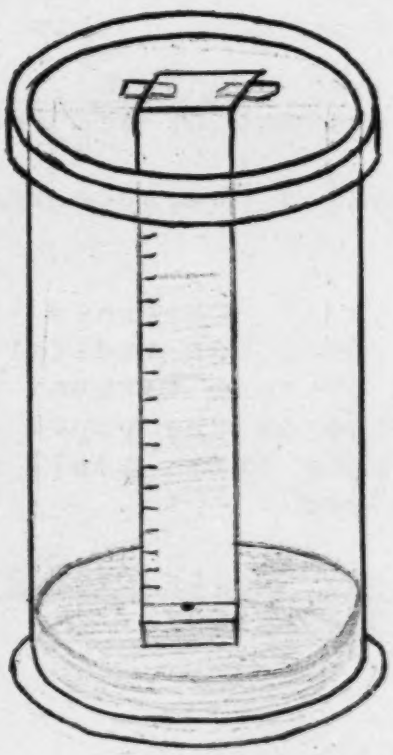


Fig. 5 Paper chromatographic set-up

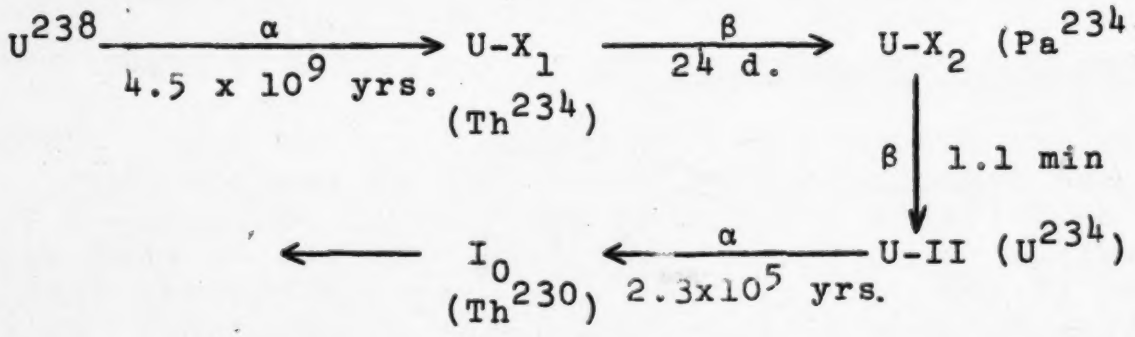
5. The strip is dried under the lamp and sprayed with a solution of ammonium molybdate to give a yellow spot for carrier. The strip is then dried again and is cut into 1 cm pieces as far as the alcohol front and each piece is counted using a G.M.-counter. The counting rates are plotted in the form of a histogram. Find R_f-factor. Alternatively, the radioactive spot may be located by autoradiography (see later).

If time of observation is available this experiment can be extended to illustrate the separation of $^{32}PO_4^{3-}$ from $^{35}SO_4^{2-}$. This proved a useful method since S^{35} is the most probable contaminating nuclide in the preparation of P^{32} by the production process: $S^{32}(n,p)\ P^{32}$.

Unfortunately, the R_f-factors of PO_4^{3-} and SO_4^{2-} are very close, hence the time needed for the separation by paper chromatographic method is about 22 hours. A 35 cm long x 3 cm wide paper is used while thesolvent is 7.5% vol/vol nitric acid in butanol.

Experiment C.1.2 Separation of Uranium Decay Chain Isotopes

The beginning of the uranium disintegration series is as follows:



If a uranium compound has not been chemically treated for several months U^{238} and U-X_1 (Th^{234}) will be in secular equilibrium (see page 7) and may be separated by suitable methods one of which is ascending paper chromatographic method.

Owing to the low energy of the β-particles emitted by U-X_1, this nuclide is counted by allowing its daughter product U-X_2 to come to secular equilibrium and counting the energetic β-particle from this. Fortunately, the half-life of U-X_2 is short (approximately 1 minute) and the equilibrium is quickly established within 10 minutes.

Material Required

Apparatus - the same as in experiment C.1.1
Saturated uranyl nitrate solution
Hexane or butanol
Acetone
5% potassium ferrocyanide solution

Procedure

The general procedure is similar to that of experiment C.1.1. Enough uranium nitrate solution is applied on to the origin of the chromatogram (500-600 counts per minute total) dropwise (dried after the application of the each drop). The chromatogram is then allowed to develop for 3 hours in 1:1 hexane-acetone mixture or about 2 hours in 1:1 n-butanol-acetone mixture. The chromatogram is dried and sprayed with 5% potassium ferrocyanide solution, a dark brown spot appears near the solvent front due to U^{238}. Using a G.M.-counter the low penetrating α-particles emitted by U^{238} are not detected, whereas radio chromatogram scanning detects the thorium spot near the base line. Since half-life of Th^{234} is 24 days, this can be checked by scanning the strip 24 days later, the thorium activity will decrease by half of the original value.

It is obviously seen from the experiment C.1.1 that a longer strip of paper and a longer time allowed for the ascending of the solvent facilitate the clear separation of the phosphate and the sulphate and this is also true for this separation. The hexane-acetone system provides a cleaner separation than the n-butanol-acetone system. On the other hands, the latter one has the advantages of being a cheaper and a faster separation system.

Experiment C.1.3 Autoradiography

In the preceeding experiments the ions under study are colourless and hence producing "invisible chromatograms". With the aid of suitable developing agents such as ammonium molybdate and potassium ferrocyanide solutions they can be made visible. In addition, if the ions are labelled with radioactive isotopes, it is possible to show up their positions by counting. Alternatively, use may be made of the power of the radiation to blacken a photographic emulsion; under a suitable conditions this will indicate the exact distribution of the radioactive material although it will not identify the nuclides concerned.

Material Required

X-ray Film
Developer
Fixer
Dark room

Procedure

Steps 1 to 4 in experiment C.1.1 are repeated. The chromatogram is dried and a drop of active phosphate solution is added on the origin and is dried (this to ascertain that the base line would show on the developed film). The chromatogram is now placed between 2 pieces of cellophane and laid on a strip of x-ray film and held securely by weights in complete darkness for an appropriate time (the usual exposure time for P^{32} is 2-3 hours and longer time is needed for weak emitters such as S^{35} or C^{14}). On

subsequent development of the film, using conventional reagents*, regions corresponding to localisation of radioactive elements are darkened so that photographic image of the tracer distribution is obtained. The experiment illustrate that radiations can blacken the film emulsion.

A indoor plant such as "scindapsus aureus" growing in a solution of ammonium phosphate labelled with P^{32} for 24 hours (dilte solution of ammonium phosphate must be used to prevent osmosis taking place) is a fine object to be autoradiographed and the image on the film provides the evidence that phosphate absorbed through roots. Alternatively, some letters can be written on a piece of filter paper using solutions of radioactive isotopes as ink and autoradiographed. Any objects can be autoradiographed providing that they are radioactive and emit strong enough radiations.

Note that in paper chromatographic methods, separation can be carried out using very minute quantity of the sample i.e. only 1-2 drops are usually sufficient.

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* Normal procedure is as follows:

1. Develop for 4-5 minutes at 25°C in developer
2. Rinse in cold water (preferably distilled water)
3. Fix for 15 minutes or longer in fixer
4. Wash in running water for 1 hour or longer
5. Hang up to dry

Developer

Metol	2g
Sodium sulphite	150g
Hydroquinone	8g
Sodium carbonate	100g
Potassium bromide	5g
Water up to	1 litre

Fixer

Sodium thiosulphate	300g
Potassium metabisulphite	25g
Chrome alum (chromium potassium sulphate)	12.5g
Glacial acetic acid	2.5ml
Water up to	1 litre

N.B. 1. Ready made developer and fixer are commercially available with the purchase of the x-ray film.

2. Developer and fixer are prepared according to what is given in "Radioisotope Laboratory Techniques" by Faires F.A. and Parks. B.H., George Newnes Limited, London (1960).

C.2 Ion Exchange

Ion exchange methods are widely used in preparation and chemical analysis. Because of its great simplicity, the ion exchange method is also becoming very common in radiochemical investigation, not only as a method of separation but also for other purposes. It has been developed over a whole range of elements with and without carriers. Basically, an ion exchanger may be considered similar to an insoluble salt, acid or base in which one of the ions is freely mobile and the other fixed. The material is termed a cation (electropositive) or an anion (electronegative) exchanger according to the sign of the mobile ion. The mobile ion may be exchanged, under suitable conditions for other ions of the same polarity, when the exchanger is treated with a solution containing these ions. Equilibrium is established between the mobile ions of the exchanger and the ions in the mixture. The retained ions are separated by washing repeatedly with a suitable eluent; those ions which are adsorbed least strongly moving down most rapidly. In practice, a column technique is almost always used.

$U\text{-}X_1$ (Th^{234}) may be extracted from uranium on a cation exchange resin. If a solution of uranyl nitrate is poured through a small column of strong cation exchange resin (such as Zeokarb-225), the $U\text{-}X_1$ is more strongly adsorbed than the hexavalent uranium. The experiment below was developed at the Isotope School, A.E.R.E., England.

Experiment C.2.1 The Separation of Carrier Free $U\text{-}X_2$ by Ion Exchange

Materials Required

Cation exchange resin (Zeokarb-225 or similar)
2.5M hydrochloric acid
0.3M sulphuric acid
12% uranyl nitrate solution
Stand with clamps
Cylindrical separating funnel (capacity of about 10-20 ml) or an ion exchange column with a jet, rubber connector and screw clip
Petri dish
G.M.-counter (coupled with a scaling unit or a rate meter)

Procedure

An ion exchange column is prepared as follows:
A small amount of cation exchange resin, stored in 2.5M hydrochloric acid or 1M sodium chloride solution (to ensure it is in H^+ or Na^+ form), is made into a slurry with distilled water and poured into the cylindrical separating funnel which is plugged with a few glass beads and glass wool. The tap of the funnel should be closed during this operation. The resin is allowed to settle and the water run off until the level is about 2 cm. above the top of the resin. This is repeated, if necessary, until the column of resin is approximately 8-10 cm deep. Wash column with about 100 ml of

distilled water (the column must not be allowed to go dry during the whole procedure.

N.B. If an ion exchange column is used, the length of the column must be increased slightly in order to increase the separation efficiency and the use of a large column is advisable due to the large amount of ions present.

A 25 ml of 12% uranyl nitrate solution is run through a column of resin at about 2 drops/second into a Petri dish mounted at an angle so that the effluent liquid may be monitored by a G.M.-counter before running over the edge into a beaker (Fig. 6). Elution with 100 ml of 0.3M sulphuric acid will remove most of the uranium but little of $U\text{-}X_1$. The presence of uranayl ion in the effluent can be checked using potassium ferrocyanide solution (red colour). After a lapse of 5-6 months the radiochemical equilibrium between uranium and its daughters - $U\text{-}X_1$, $U\text{-}X_2$ is reached and can be used again as a source of $U\text{-}X_1$ or $U\text{-}X_2$.

To extract $U\text{-}X_2$ (Pa^{234}) from $U\text{-}X_1$ (Th^{234}), simply run 2.5M hydrochloric acid through the column. The effluent is monitored by a G.M.-counter and its activity will be seen to increase up to a maximum. At this point the flow of HCl is stopped and readings of the ratemeter are taken at 10-20 second intervals or 10 seconds counts are taken every 20 seconds using a scaling unit. The half life of Pa^{234} is therefore determined either by observation or by plotting a decay curve if the decrease in count rate with respect to time is recorded. Literature value of the half-life of Pa^{234} is 1.14 minutes.

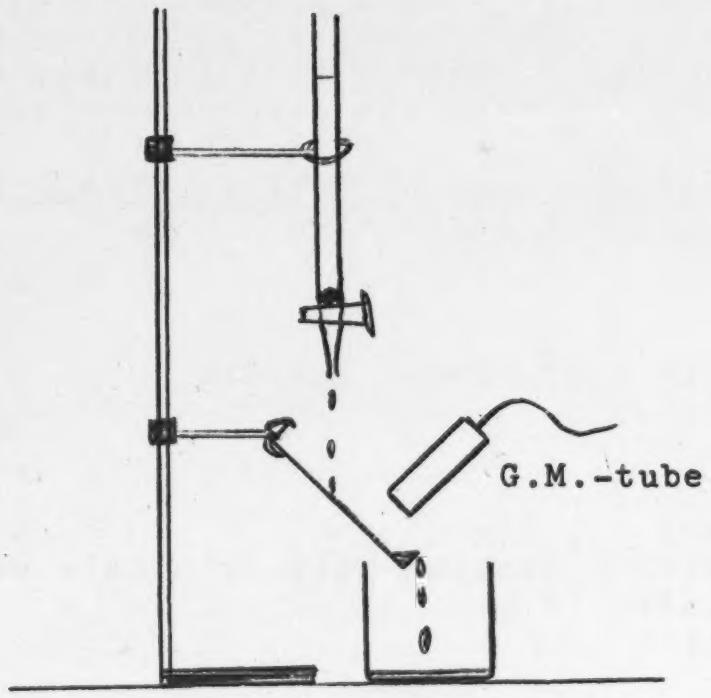

Fig. 6 Ion exchange set-up

C.3 Solvent Extraction

The solvent extraction method is a powerful method of separation and should prove extremely useful in the separation of tracer quantities particularly where speed of separation is required such as in radiochemical work involving a short lived nuclide.

In the separation by solvent extraction, advantage is taken of the fact that, if a substance is soluble in two solvents which are immiscible, it will distribute itself between the two phases. When equilibrium is reached the ratio of the concentration of the substance in the two phases is called the "distribution ratio" or "partition coefficient" which is a constant at a given temperature and is independent of concentration. Usually in practice, the substance in question is dissolved in a particular solvent, generally water. A quantity of the immiscible extractant is added to this solution, the two are shaken together, and then separated. This may need repetition, depending on the completeness of extraction desired and the distribution constant. There are, however two aspects of solvent extraction techniques which find wide application in radiochemistry. The first of these is the use of a specific complexing agent in order to render the desired constituent soluble in a particular solvent. The second method is by careful control of pH, with which it is possible to achieve rather complete separations of the desired material from many other ions present. There are many types of apparatus used depending on the size of the operation. The simplest one is the shaking of two phases together in a separating funnel (with pressure frequently and carefully released by the removal of the stopper). The two phases are then separated by drawing off the bottom layer via the tap.

Since the cupferron-complex of protactinium was found to be extracted into benzene, chloroform, diethyl ether, amyl acetate, methyl isobutyl ketone, isobutyl ketone and nitromethane from aqueous solution, ranging from pH 1 to 4 in acid, hence solvent extraction of $U\text{-}X_2$ (Pa^{234}) from its parents was used satisfactorily.

Experiment C.3.1 The Extraction of Carrier Free $U\text{-}X_2$ by Solvent Extraction

Material required

Saturated solution of uranyl nitrate
2% cupferron solution
6M nitric acid
Amyl acetate
Stand with clamp
G.M.-counter with a scaling unit or a rate meter
Measuring cylinder 10 ml
Separating funnel

Procedure*

Take 10 ml of saturated uranyl nitrate solution (measured with a 10ml cylinder) in a separating funnel. This is acidified with 3 ml. concentrated nitric acid. Add 6 ml of 2% cupferron solution in 10 ml amyl acetate. Shake thoroughly and quickly (since U-X_2 has a very short half-life) to extract U-X_2 complex into the amyl acetate layer. Allow to settle, then the bottom layer (aqueous layer) is quickly separed, the organic layer is run slowly into a petri dish (see the setting of the apparatus from Expt. C.2.1) and is monitored for the decay of U-X_2 in the same manner as described in experiment C.2.1.

In place of amyl acetate, other solvents mentioned prior to commencing of this experiment can be used with slight modification for example the extraction of U-X_2 from a hydrochloric acid solution of uranyl nitrate into isobutyl ketone should yield a similar result to that discussed above.

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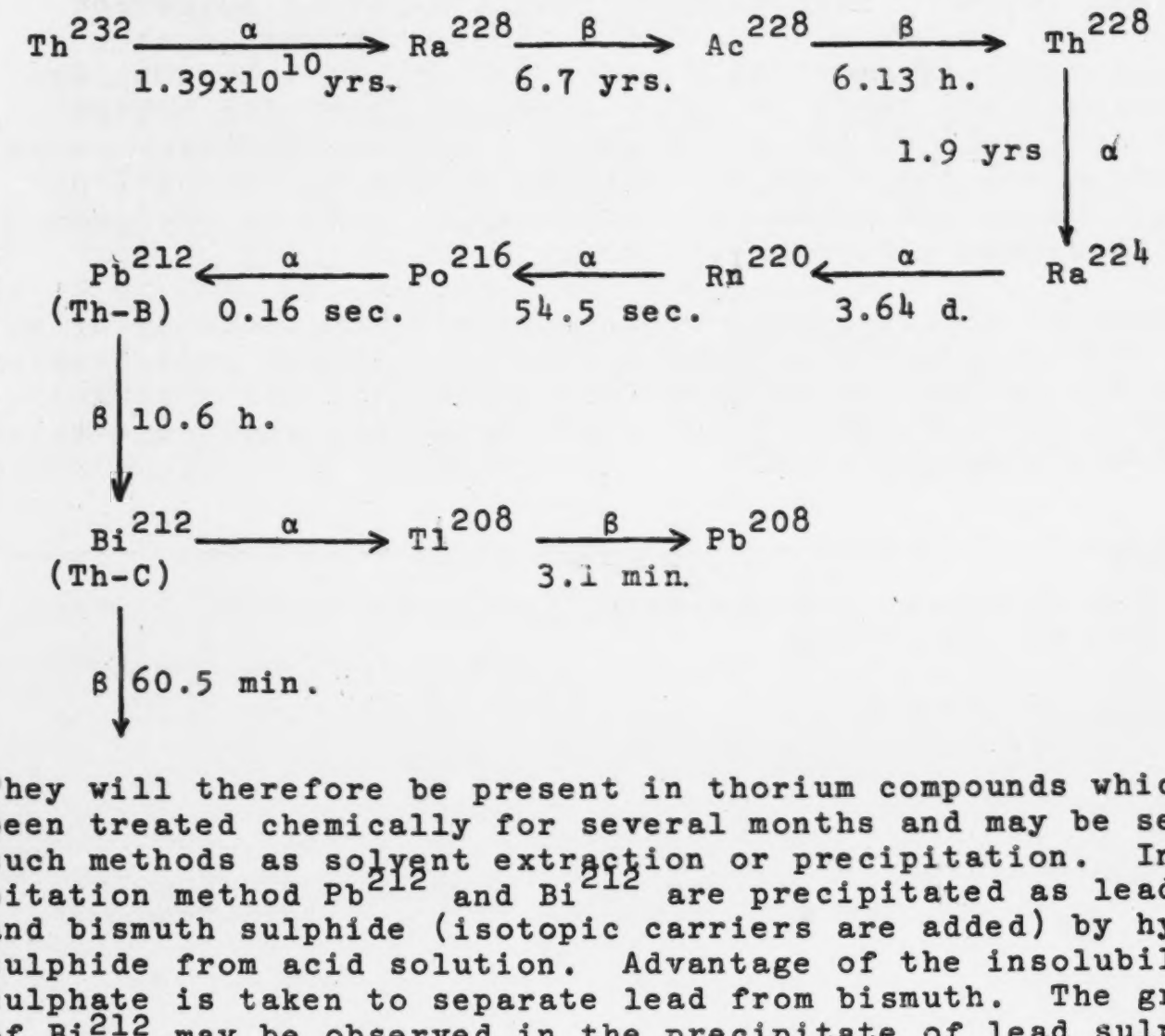
*Broadbank R.W.C, and S. Dhabanandana, Letter to editor, J. Chem. Ed., Vol. 37, No. 3, (1960).

C.4 Precipitation

Thorium-B (Pb^{212}) and thorium-C (Bi^{212}) are consecutive members of the thorium disintegration series.



They will therefore be present in thorium compounds which have not been treated chemically for several months and may be separated by such methods as solvent extraction or precipitation. In the precipitation method Pb^{212} and Bi^{212} are precipitated as lead sulphide and bismuth sulphide (isotopic carriers are added) by hydrogen sulphide from acid solution. Advantage of the insolubility of lead sulphate is taken to separate lead from bismuth. The growth curve of Bi^{212} may be observed in the precipitate of lead sulphate (takes approximately 4 hours to grow to a maximum thereafter it will decay with the half life of Pb^{212}). The decay of Bi^{212} (Th-C) is followed in the solution of sulphate of bismuth.

Experiment C.4.1 Separation of Th-B and Th-C by Precipitation

Material Required

Thorium nitrate
2M hydrochloric acid
1.5%bismuth chloride solution
1.5% lead nitrate solution
Hydrogen sulphide gas
G.M.-counter with a scaling unit or a rate meter

Procedure

A dilute acid solution of thorium nitrate is prepared by dissolving approximately 10 grams of thorium nitrate in a little water and a few drops of 2M hydrochloric acid are added. Approximately 1 ml of 1.5% solution of lead nitrate and 0.5 ml of 1.5% solution of bismuth chloride are added as carriers of Th-B and Th-C. Hydrogen sulphide is passed into a hot solution of thorium nitrate until precipitation of sulphide is complete. The precipitate is washed with warm 1% hydrochloric acid to free it from thorium salts. The washings being separated by centrifuging and discarded. The sulphides are separated afterwards by dissolution in dilute nitric acid followed by precipitation of lead sulphate (carrying lead-212 or Th-B) having a little concentrated sulphuric acid as a precipitating agent. The tube is left in a beaker of almost boiling water. After evolution of nitrous oxide fumes has ceased, it is cooled and diluted to approximately 10 ml with water and centrifuged. The time is now noted and the solution x which contains Th-C activity is set aside while the precipitate of lead sulphate is washed with 5% sulphuric acid, water and acetone successively and is made a slurry with small amount of acetone and transferred to a source tray, is dried and covered with "Cellotape". The source is counted for activity of Th-C which grow from zero to a maximum every half an hour. The activity due to Th-B is screened with a piece of aluminium absorber of about 250 mg/cm^2 placed between the source and detector (G.M.-tube). Growth of Th-C in Th-B should be observed.

The half life of Th-C (60.5 minutes) can be determined from solution x, employing the same procedure as described in experiment C.2.1.

Th-C decays by an α emission to thallium-208 which is a β-emitter with half life of 3.1 minutes. The study of half-life can be made on Tl^{208} instead of Bi^{212}. The solution x which contain soluble sulphate of bismuth is poured through a small column of the hydroxide form of "Deacedite FF" or similar (strong anion exchange resin). Bismuth is held very strongly under these conditions and after 10 minutes or so the Tl^{208} which has grown may be washed out with very dilute hydrochloric acid into a petri dish and is monitored for the decay of Tl^{208} and hence its half-life is obtained.

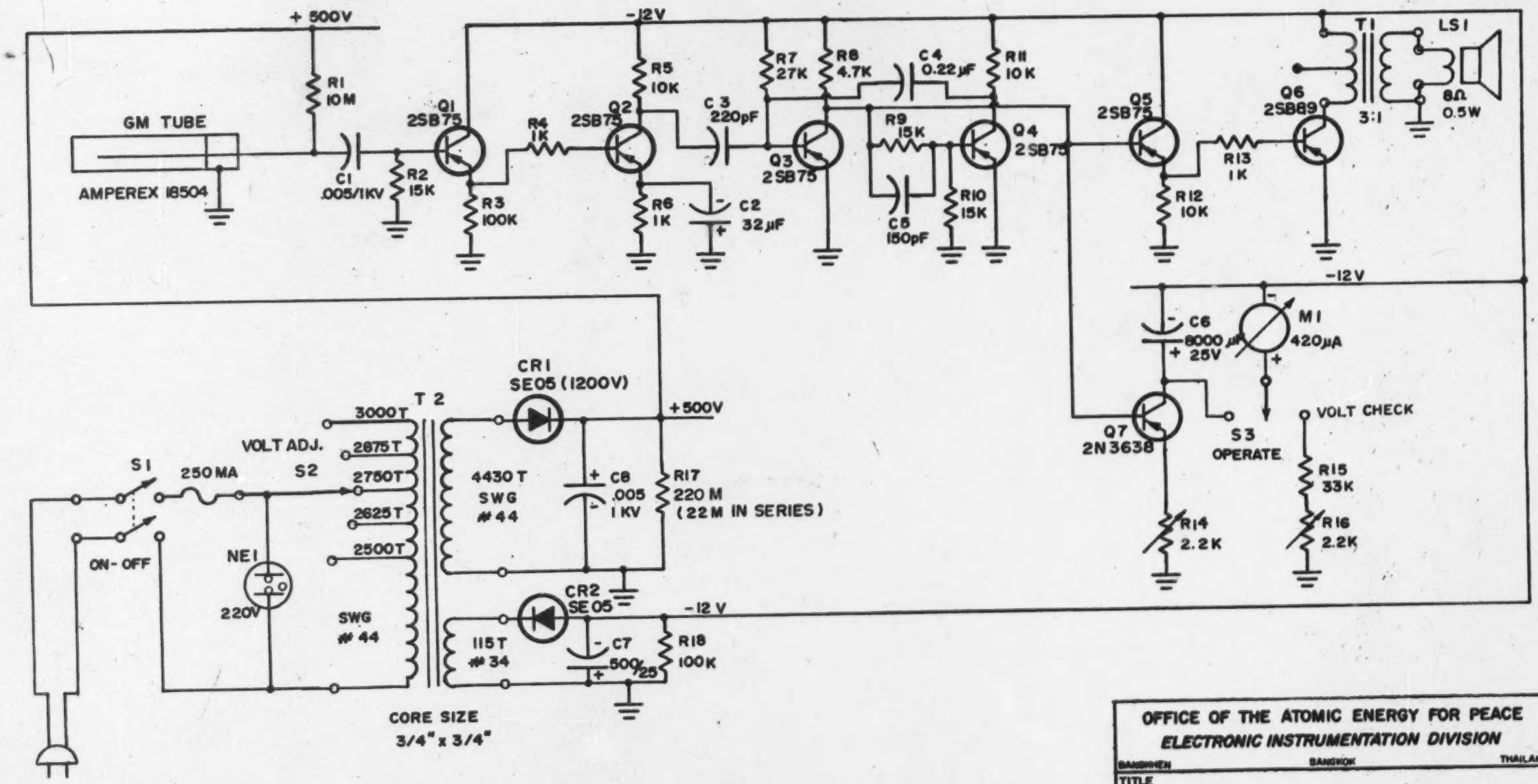
The above experiment has been developed at the Radiochemistry Section, Leicester Regional College of Technology, England.

Discussions and Conclusions

Short lived nuclides are separated from their parents by simple methods and this provides a useful demonstration of radioactive growth and decay.

The experiments present in this section except experiments C.1.1, C.1.2 and C.4.1 yield carrier free tracers. Whenever carrier is added, the amount of carrier is kept as small as possible, usually about 5-50 mg in order to minimize measurement problems.

There is no radiation hazard in any experiments if the amount of radioactive material is kept to the minimum necessary for satisfactory measurement. Nevertheless, it should be made a habit not to use mouth operation, not to pick a source of activity up with a bare hand and always use a pair of tweezer to pick a solid source and wrap a beaker or tube etc. containing radioactive substances with tissue paper before holding by hand. Uranium solutions are toxic. There is no danger to health arising from the radioactive substances but there is appreciable risk of contaminating the counting apparatus.



OFFICE OF THE ATOMIC ENERGY FOR PEACE
ELECTRONIC INSTRUMENTATION DIVISION
BANGKHEN BANGKOK THAILAND

TITLE

TRAINING RATEMETER MODEL 1503 A

Aims and Objectives of this Project

The General Conference of Unesco, at its thirteenth session, adopted Resolution 2.122 to organize a Pilot Project for Chemistry Teaching in Asia for the purpose of initiating a fundamental re-orientation in the way of teaching chemistry through the use of modern technical devices and methodology. An agreement was signed between the Government of Thailand and Unesco on 13 July 1965 to locate the Project at Chulalongkorn University, Bangkok. The Project started as a regional project. In addition to its regional activities, the Project centre has increasingly served as a national centre for Thai science education.

The primary aim of the Project is to assist science educators in Asia in their task of carrying out reform of chemistry teaching. The Project is operating along two major lines which are distinct but co-ordinated:

1. Modernization of the chemistry courses and development of new teaching materials.
2. Assistance in carrying out in-service and pre-service teacher training, improvement of examinations and use of the latest methods of teaching.

Science educators in Asia may wish to request some or all of the following resource materials (at no cost) in sample quantities to help them carry out curriculum reform:

1. Programmed Instruction Sequence, 1966.
2. Teachers' Guide to the above programmed sequence, 1966.
3. 8 mm. Film Loops in Cassettes.*
4. Film Loop Production Notes, 1967.
5. Teachers' Guide to Film Loops, 1967
6. Compound Formation (Vols, I and II), Teachers' Digest, 1967, 1968.
7. Chemical Equilibria, A Teachers' Digest, 1968.
8. Experiments on Chemical Equilibria, 1968.
9. Experiments on Compound Formation, 1969.
10. Compound Formation Vol. 1 (Thai translation), 1969.
11. Experiments on Chemical Equilibria (Thai translation), 1969.
12. Newsletter, a bi-monthly periodical.
13. Prototypes of low cost kits:** "Teaching Experiments on Chemical Equilibria", "Teaching Experiments on Compound Formation" and "Teaching Experiments on Rate of Chemical Reactions."
14. Experiments on Rate of Chemical Reactions, 1969.
15. Experiments on Nuclear Science, 1969.

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* Available at a cost of U.S. $ 6.00 per film loop.
** Cannot be supplied outside Thailand.